To etch or not to etch? This is no longer the question.

Self-Etch and Etch&Rinse Adhesive
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1 Introduction

It is very often assumed that modern adhesive dentistry was initiated and started by Buonocore in the 1950s who modified enamel by etching with phosphoric etching followed by a rinsing step – nowadays referred to as etch&rinse mode. However, already 1949 an acid methacrylate monomer was developed by Hagger and used in the product Sevriton Cavity Seal which was able to interact with dentin – laying the foundation for self-etching adhesives. This and more details on how adhesion to enamel and dentin developed over time and how different approaches were followed are described elsewhere (Söderholm, 2007).

Still today the discussion in the academic world continues whether etch&rinse (E&R) (e.g. etching with phosphoric acid followed by a rinsing step) or self-etch (SE) is the better approach to achieve adhesive restorations with high longevity. Besides fundamental considerations, there are clinical aspects making both approaches attractive and useful under specific circumstances.

There is no doubt that adhesion to enamel is much easier to establish in the E&R mode. And that the SE mode offers a lot of clinical advantages while bonding to dentin. That might be the reason why Selective Enamel Etching (SEE) (e.g. etching enamel with phosphoric acid while using self-etch mode for dentin) is getting more and more popular as it combines advantages from both etching modes.

Furthermore, final outcome in dentistry is highly dependent on the operator. Using only few products with detailed knowledge how to best handle and to apply with perfection will improve overall quality.

Thus, the goal of developing Xeno Select was to provide an adhesive that can be used purely as E&R, SE on both enamel and dentin as well as in SEE mode to reduce the needed number of adhesives to cover all etching modes.

1.1 Storage conditions and shelf-life

In general, polymerizable acrylate monomers are used in an aqueous, acidic solution as this is a requirement for one component dental adhesives usable in self-etch mode. These monomers have to face two different potential degradation reactions:
First, reactive solvent molecules can add at the polymerizable group (Michael-addition), which results in the loss of its polymerizable function, although the overall monomer structure is not disrupted (Figure 1).

![Figure 1](image1.png)

**Figure 1** Addition of solvent molecules (i.e. ethanol) to polymerizable groups (grey) results in loss of the polymerizable function (open circle) ($X = O$ for ester; $X = NH$ for amide)

Second, the reaction of water with a polymerizable group does not destroy its function, but separates the polymerizable group from the rest of the monomer by hydrolysis (Figure 2a).

![Figure 2a](image2a.png)

**Figure 2a** ester linkage of polymerizable group

![Figure 2b](image2b.png)

**Figure 2b** ester linkage of polymerizable group with phosphoric acid function

**Figure 2** Separation of polymerizable groups (grey) by hydrolysis

Both degradation mechanisms result in a reduced density of the polymer network. In the case of functionalized acidic monomers the hydrolysis may lead to acidic components that are no longer polymerized into the network (Figure 2b). Thus, further undesired etching by these mobile groups could occur at the interface.

Separating relevant components by primary packaging with two compartments or in two separate bottles is one way how to achieve sufficient storage stability at room temperature over the respective shelf-life.

However, even certain two bottle self-etching adhesives need to be stored at lower temperature implying that stability of the chosen chemistry is not guaranteed at room temperature (Table 1).
### Table 1

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Storage condition</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clearfil SE Bond</td>
<td>2 – 8 °C</td>
<td></td>
</tr>
<tr>
<td>Optibond XTR</td>
<td>2 – 8 °C</td>
<td></td>
</tr>
<tr>
<td>Xeno III</td>
<td>2 – 24 °C</td>
<td></td>
</tr>
</tbody>
</table>

Storage conditions of two-component self-etching adhesives

Most one-component formulations as listed in Table 2, cannot be stored at room temperature for their entire shelf-life.

### Table 2

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Storage condition</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>AdheSE One F</td>
<td>2 – 28 °C</td>
<td></td>
</tr>
<tr>
<td>Bond Force</td>
<td>0 – 10 °C</td>
<td></td>
</tr>
<tr>
<td>Clearfil S3 BOND Plus</td>
<td>2 – 8 °C</td>
<td>when not in use</td>
</tr>
<tr>
<td>Gænial BOND</td>
<td>1 – 28 °C</td>
<td>when not in use for a prolonged period of time, store in refrigerator</td>
</tr>
<tr>
<td>iBOND Self Etch</td>
<td>-10 – 25 °C</td>
<td></td>
</tr>
<tr>
<td>OptiBond All-In-One</td>
<td>2 – 8 °C</td>
<td></td>
</tr>
<tr>
<td>Scotchbond Universal</td>
<td>2 – 25 °C</td>
<td></td>
</tr>
</tbody>
</table>

Storage conditions for one-component self-etching adhesives according to the respective manufacturer instructions as of 2014-01-13

To combine ease of use of one-component formulations, self-etching technology, and storage at room temperature for the entire shelf-life, monomers and formulations are needed as described in Chemistry.

## 2 Xeno Select – Product Description

Xeno Select is a one component dental adhesive designed to bond resin-based, light-curing restorative materials to enamel and dentin.

It can be used in all etching techniques:
- Self-Etch
- Etch&Rinse
- Selective Enamel Etching
Xeno Select provides high application security even on over dried dentin. This results in a consistent, reliable performance and virtually no postoperative sensitivities. The acetone-free composition allows chairside storage at room temperature and 30 minutes working time in a closed CliXdish™.

2.1 Chemistry

The high tolerance of Xeno Select towards storage conditions of up to 24 °C is obtained by using following monomers

- acryl resins with amide groups instead of ester groups
- *inverse* functionalized phosphoric acid esters instead of ester functionalized phosphoric acid esters

and

- tertiary butanol instead of lower molecular weight alcohols, like ethanol, as a solvent

The Xeno Select components and their functions are given in more detail in Table 3. Their principal chemical structure is shown in Figure 3 and Figure 5.

<table>
<thead>
<tr>
<th>Component</th>
<th>Function</th>
</tr>
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<tbody>
<tr>
<td>Bifunctional acryl resin with amide functions</td>
<td>Crosslinker</td>
</tr>
<tr>
<td>Acryloylamino alkylsulfonic acid</td>
<td>Etchant and wetting aid</td>
</tr>
<tr>
<td>“inverse” functionalized phosphoric acid ester</td>
<td>Etchant, adhesion promoter and wetting aid</td>
</tr>
<tr>
<td>Camphorquinone, Coinitiator</td>
<td>Photoinitiator system</td>
</tr>
<tr>
<td>Butylated benzenediol</td>
<td>Stabilizes monomers during storage</td>
</tr>
<tr>
<td>Water</td>
<td>Solvent for the resins and etch promoter</td>
</tr>
<tr>
<td>tert-Butanol</td>
<td>Solvent for the resins and mild stabilizer</td>
</tr>
</tbody>
</table>

Table 3 Components of Xeno Select and their function
In Xeno Select tert-butanol is used as solvent. The alcohol group in tert-butanol is shielded by the surrounding methyl groups of the tertiary group (Figure 3). Therefore tert-butanol is not able to chemically react by addition at the acrylate resins in the same way ethanol and iso-propanol do at low pH (Figure 4). For this reason, tert-butanol was chosen for Xeno Select to ensure a chemically stable formulation.

Xeno Select comprises proprietary monomers as schematically described in Figure 5. The difference to conventional monomers is the introduction of amide groups and “inverse” methacrylic ester functions instead of ester functions.
Based on their molecular structure, amides are more stable in an aqueous, acidic solvent solution compared to ester. This protective effect is illustrated in Figure 6 and the resulting stability is demonstrated in Figure 7.

In addition to amide monomers, “inverse” functionalized monomers are used in Xeno Select. These monomers are characterized by the attachment of the polymerizable methacrylate function to the acidic phosphoric acid ester function by a more hydrolysis-stable ether bond instead of an ester bond (Figure 8).
2.1.1 Summary of Xeno Select chemistry

The use of tert-butanol as part of the solvent, combined with acryl resins with amide functions, and “inverse” functionalization guarantees that polymerizable functions remain available to form a polymer network for the entire shelf-life even in a highly acidic aqueous one-component formulation.

2.2 Components and functions

Besides their stability in an aqueous, acidic environment, each ingredient of Xeno Select has to fulfil a specific function to guarantee the overall success of Xeno Select.

As crosslinkers, the bifunctional acryl resins with amide functions ensure the formation of a dense resin network upon light-curing.

The “inverse” functionalized acidic monomer used in Xeno Select comprises a phosphoric acid ester group (Figure 8). It has been demonstrated by μRAMAN spectroscopy that such groups promote chemical interaction between monomers and tooth substance (Latta, 2007).

Figure 8 Ester functionalization (a) compared to “inverse” functionalization (b)

Figure 9 Possible interaction of phosphoric acid ester group with tooth substrate.
The second acidic monomer, the acryloylamino alkylsulfonic acid, was added to the formulation to adjust the acidity of Xeno Select and to ensure the formation of a well-defined etch pattern as described in the chapter Micro Morphology.

The use of a water/tert-butanol solvent solution provides the formulation with a well-balanced polarity. On one hand the polarity is high enough for the acidic monomers to be effective. On the other hand the polarity also allows less polar ingredients, like the initiators, to dissolve.

In addition, the low volatility of the water/tert-butanol solvent solution makes it possible to use Xeno Select from closed CliXdish™ for up to 30 minutes, making it more convenient in daily practice.
3 In vitro Investigations

As the final clinical performance of a dental adhesive highly depends on the operator and other influencing factors, a first step in evaluating a new adhesive should be in vitro testing under conditions as controlled as possible in order to allow comparison to controls. Furthermore, there is no single in vitro method allowing a conclusive prognosis of the clinical performance. Therefore, a comprehensive test program was conducted in order to evaluate the performance of Xeno Select. This included shear bond strength (SBS) to enamel and dentin, micro morphology, marginal integrity, and dye penetration. Xeno Select was compared to Xeno V+ and Scotchbond Universal in self-etch mode and to the latter adhesive in etch&rinse mode as well.

3.1 Shear bond strength to enamel and dentin

(Mark A. Latta, Omaha (NE), USA)
Shear bond strength (SBS) was not only tested under standard conditions but substrate relevant variations were included as well. Enamel was either roughened with 600 grit sand paper or left unprepared and dentin bonding was tested either under ideal conditions (e.g. moist dentin) or challenged by different degrees of moisture. Over dried dentin was achieved by air blow for 10 seconds or over wet dentin by application of 2.5 µl water over an area of 4 mm diameter.
Adhesives were applied and light cured before positioned in an Ultradent jig. Spectrum TPH3 was immediately applied and light cured through the respective mold.
Specimens (n=12) were stored for 24 hours at 37°C before and after thermo cycling which comprised 6,000 cycles 5-55°C with a dwell time of 20 seconds. Cross head speed for debonding was 1 mm/min.

3.1.1 SBS to enamel – prepared and unprepared

SBS of Xeno Select to enamel was equally high independent of the etch mode applied (see Figure 10). However, in etch&rinse mode Xeno Select showed higher bond strength compared to Xeno V+ in self-etch mode.
In esthetically demanding class III, class IV, and class V restorations very often a smooth bevel is prepared to mask the enamel composite margin. However, this makes it difficult to fully remove excess material beyond the prep line. After using self-etching adhesives it is often reported that either marginal staining under these thin feather edges or chipping occurs making a refurbish (e.g. polishing) necessary. Therefore, adhesion to unprepared enamel was tested as well (see Figure 11).

**Figure 10** Shear Bond Strength to enamel in self-etch and etch&rinse mode after 6000 thermo cycles.

**Figure 11** Shear Bond Strength in both etching modes to enamel being roughened with 600 grit or left unprepared.

SBS of Xeno Select in etch&rinse mode to unprepared enamel was comparable to prepared enamel whereas in self-etch mode SBS to unprepared enamel was lower compared to prepared enamel. Therefore, Xeno Select offers the possibility to improve adhesion to unprepared enamel when combined with separate etching (e.g. selective enamel etching (SEE)).
3.1.2 Dentin bonding – robustness towards degree of dentin moisture

Using a protocol originally designed to challenge etch&rinse adhesives (Latta et al., 2010), Xeno Select was compared to another universal-etch adhesive when applied to dentin with three different degrees of moisture.

Figure 12  SBS to dentin with different degrees of moisture.

Figure 12 shows that SBS of Xeno Select to over dried dentin is higher compared to the control in both etching modes. To moist and over wet dentin Xeno Select provides higher bond strength compared to the control in etch&rinse mode, only.

3.1.3 Compilation of SBS to enamel and dentin

A compilation of SBS values obtained by the same operator using the identical protocol is shown in Figure 13 and demonstrates the high level of adhesion to dentin and enamel achieved by Xeno Select compared to self-etch adhesives.

Figure 13  Compilation of shear bond strength values to roughened enamel and moist dentin after 6000 thermo cycles.
3.2 Micro Morphology

(Jorge Perdigão, Minneapolis (MN), USA)

In order to visualize the capability to properly wet and infiltrate different substrate surfaces Scanning Electron Microscopy (SEM) was applied to investigate interfaces either directly or via a replica technique.

3.2.1 Etching pattern and wetting of enamel

Enamel specimens were roughened with a course diamond bur or left unprepared. After application of Xeno Select specimens were immersed in acetone for 24 h in a rotator to dissolve the resin or were build-up with a flowable composite to replicate the adhesive enamel interface.

The investigator summarized the findings in self-etch mode as following (see Figure 14).

"Some areas, especially in the roughened enamel specimens, showed an etching pattern characteristic of interprismatic etching, similar, but not as deep, as phosphoric acid etching. Other areas showed a pattern of intraprismatic etching, which would be expected from a self-etching adhesive."

![Figure 14](image)

**Figure 14** Etching pattern on enamel – influence from roughening.

As expected, etching pattern in etch&rinse mode is much more pronounced and Xeno Select showed very good wetting and penetration of these fine patterns as can be seen from respective replicas (Figure 15).
Findings from replicas were summarized by the investigator as follows:

"The etching pattern and adhesive penetration into both roughened and not roughened etched enamel was consistent with the patterns we have observed for total-etch adhesives, such as XP Bond - deep interprismatic dissolution and profuse nano-porosities within the intraprismatic area."

3.2.2 Hybrid layer of moist vs. over dried dentin

Post-operative sensitivity (POS) is a commonly reported issue by general dental practitioners when placing posterior composite restorations. It is believed that incomplete infiltration or sealing of dentin is one of the critical conditions leading to this phenomenon. Etching dentin results in denuded collagen fibers which are prone to collapse and stick to each other resulting in a difficult to penetrate mesh and an incomplete hybrid layer.

Therefore, Xeno Select was investigated regarding hybrid layer formation on moist and over dried dentin.
The applied etching mode had a significant influence on the hybrid layer thickness.

- self-etch: 0.4-0.6 μm
- etch&rinse: 3.0-4.0 μm; with abundant resin tags

More important was the finding that hybrid layer formation was not affected by the degree of moisture as stipulated by the investigator:

"None of the specimens showed any gap or any area of deficient infiltration of the demineralized collagen layer, even after deproteinization in sodium hypochlorite"

These findings from the micro morphological investigation support the robust SBS data when dentin was over dried (see Figure 12).

3.3 Marginal integrity and dentin sealing

Clinical marginal integrity is claimed to be predictable by in vitro chewing simulation (Frankenberger et al., 2007). Though there is no correlation to clinical outcome published, using dye penetration to evaluate dentin sealing of the cavity floor allows differentiating among adhesives and/or etching modes (Rosales Leal, 2007). Therefore, chewing simulation in class II and dye penetration in class V were investigated.

3.3.1 Marginal integrity of class II restorations after chewing simulation

(Roland Frankenberger, Marburg, Germany)

Standardized class II cavities were prepared and restored before thermo-mechanical loading was applied using parameters shown in Figure 17.
Marginal integrity as seen under SEM at x200 magnification and expressed as percentage of perfect margins before and after thermo mechanical loading (TML) is shown in Figure 18.

For Xeno Select as well as for the respective control it was found that application of the adhesive in etch&rinse mode led to significantly better marginal integrity in enamel after TML. However, in dentin etching mode had no influence on the outcome. These findings support using selective enamel etching (SEE) when dentin margins are i.e. difficult to rinse.

Figure 18 Marginal integrity before and after thermo mechanical loading (TML)

3.3.2 Dye penetration in class V restorations after thermo cycling

(Juan Ignacio Rosales Leal, Granada, Spain)

Two class V restorations were placed in five 3\textsuperscript{rd} molar teeth per group and stored for 24 hours at 37°C before thermo cycled 4000 times between 5-55°C with a dwell time of 30 seconds. Afterwards teeth were immersed in a 0.5% water solution of basic fuchsin for 24 h and rinsed for 5 min with water. Three slices of 1 mm thickness
were obtained from each specimen by cutting in *bucco-lingual* direction and dye penetration along the composite-cavity interface was measured and is expressed as percentage of the total interface length in Figure 19.

In order to allow direct comparison between the used adhesives Ceram•X mono+ was used as composite for all restorations.

**Figure 19** Percentage of dye penetration in class V restorations.

Xeno Select showed better sealing of dentin margins in both etching modes compared to control adhesives.

Enamel margins didn't show dye penetration when etch&rinse mode was applied.

Dye penetration allows not only visualizing whether dye penetrates between composite and cavity walls, it also visualizes whether dentin tubules are sealed despite a defective composite dentin interface or are permeable so that dye may infiltrate.

**Figure 20** Percentage of slices showing dye permeability into dentin tubules.

In self-etch mode none of the tested adhesives showed any dentin tubule dye permeability.
Both universal-etch adhesives showed only slight dentin tubule dye permeability under etch&rinse mode which was less compared to most etch&rinse adhesives as published earlier (Rosales Leal, 2007) and shown for a selection of etch&rinse adhesives in Figure 21.

![Figure 21](image_url)

**Figure 21** Percentage of slices showing dye permeability into dentin tubules.

### 3.4 Summary and conclusion of in vitro studies

Xeno Select performed equal or better compared to the controls independent of the etching mode. Furthermore, it showed high robustness towards different degrees of moisture in dentin. Based on the shown in vitro data Xeno Select is a true universal-etch adhesive providing high performance and technique robustness.
4 Clinical Data

4.1 Class II study at the University of Bologna

Background
It is often discussed that dentin etching with phosphoric acid may increase the risk of postoperative hypersensitivities. Therefore, the main purpose of the study was to show that introducing a separate etching step for Xeno Select does not present an increased risk for occurrence of postoperative hypersensitivities compared to other established etch&rinse adhesives.

Methods and Materials
The study took place at the Department of Conservative Dentistry, University of Bologna, Italy, under supervision of Principal Investigator Professor Giovanni Dondi Dall'orologio, head of the Department of Conservative Dentistry.

A total of 120 Class II restorations were placed in molars and/or premolars using Xeno Select in etch&rinse mode (test restorations) or using the established etch&rinse adhesive XP Bond® (control restorations) both in combination with SDR and Ceram•X mono+.

Each of the 60 enrolled patients received one test and one control restoration in a randomized semi-blinded manner in non-adjacent teeth only, i.e. 60 restorations per adhesive were placed. All included teeth had to be free of severe preoperative hypersensitivity (pain level according to 11-point Visual Analog Scale (VAS) had to be ≤ 3; score 0 means absence of pain, score 10 means most severe pain possible).

Evaluation of incidence and degree of postoperative hypersensitivities according to VAS and of restoration failures was done 7-28 days after restoration placement for all 60 patients.
Results

1. Neither Xeno Select nor XP Bond® did cause any case of postoperative hypersensitivity.
2. No failures were observed.
3. No adverse event or other relevant clinical problem occurred.

Discussion
The early recall after restoration placement was well-suited for the evaluation of adhesive-related postoperative hypersensitivities which was the main focus of the study.

No restoration failures were observed. At the same time, the early recall is not sufficient to draw conclusions about long-term adhesive performance – but this was not the intention of the study.

Conclusions
Based on the results from the study, no additional risk regarding postoperative hypersensitivities or restoration failures in the considered recall period can be expected when using Xeno Select in etch&rinse mode instead of the established etch&rinse adhesive XP Bond®.

4.2 Clinical experience: Feedback from General Dental Practitioners

132 dentists in Germany tested Xeno Select during routine clinical use in their private offices and placed more than 6,000 restorations. The results underline the outstanding performance of Xeno Select in daily clinical settings:

- The handling of Xeno Select was appreciated by 99% of all users (48% rated it as “good”, 51% even rated it as “excellent”)
- 92% found the viscosity of Xeno Select “exactly right”
- More than 74% of the dentists even preferred Xeno Select over their previously used adhesive
- Postoperative hypersensitivities were reported in less than 0.4% of all cases
5 Instructions for Use

The up-to-date version can be found in all European languages on www.dentsply.eu.

6 References


¹ Erratum in: J Adhes Dent. 2007 Dec;9(6):546
7 Glossary and Abbreviations

IFU Instructions for Use
E&R Etch&Rinse
Etching with phosphoric acid which has to be rinsed off (formerly referred to as Total Etch Technique)
POS Post operative (hyper)sensitivity
SBS Shear Bond Strength
SE Self-Etching
SEE Selective Enamel Etching
SEM Scanning Electron Microscope
TC Thermo Cycles
TML Thermo Mechanical Loading

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Brand (abbreviation(s); Manufacturer):
AdheSE One F (AOneF; IvoclarVivadent)
Adper Prompt L-Pop (3M ESPE)
BondForce (Bforce; Tokuyama)
Clearfil SE Bond (Kuraray)
Clearfil S3 Bond (CFS3; Kuraray)
Clearfil S3 Bond Plus (Kuraray)
Gaenial Bond (Gaenial; GC)
iBOND Self Etch (iBond SE; Heraeus)
OptiBond All-in-One (OB AiO; Kerr Hawe Neos)
Scotchbond Universal (SB Universal, SBU; 3M ESPE)
Scotchbond 1 XT (3M ESPE)
Syntac Classic (Ivoclar Vivadent)
Tetric EvoCeram (Ivoclar Vivadent)
Z250 (3M ESPE)